

TOTAL PETROLEUM HYDROCARBONS (TPH) as Gasoline and Diesel

SW-846 Method 8015B (Revision 2, December 1996)

Table 1A. Summary of Contract Required Quantitation Limits, Holding Times, and Preservation for Total Petroleum Hydrocarbons (TPH) as Gasoline and Diesel

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation
Total Petroleum Hydrocarbons (TPH) as Gasoline in Water Samples	<u>Technical</u> : 7 days from collection; <u>Contract</u> : 5 days from receipt at laboratory	Cool to 4EC ±2EC
TPH as Gasoline in Water Samples	<u>Technical</u> : 14 days from collection; <u>Contract</u> : 10 days from receipt at laboratory	HCl or H ₂ SO ₄ to pH<2; Cool to 4EC ±2EC
TPH as Gasoline in Soil Samples	<u>Technical</u> : 48 hours <u>Contract</u> : 48 hours	Cool to 4EC ±2EC; sealed zero headspace containers; freezing can extend the holding time ^{b,c}
TPH as Gasoline in Soil Samples	<u>Technical</u> : 14 days from collection; <u>Contract</u> : 10 days from receipt at laboratory	Preserved samples: in methanol ^d or sodium bisulfate ^e
TPH as Diesel in Water Samples	<u>Technical to Extraction</u> : 14 days from collection; <u>Contract to Extraction</u> : 10 days from receipt at laboratory <u>Technical and Contract to Analysis</u> : 40 days from extraction	Cool to 4EC ±2EC; HCl or H ₂ SO ₄ to pH<2 ^f
TPH as Diesel in Soil Samples	<u>Technical to Extraction</u> : 14 days from collection; <u>Contract to Extraction</u> : 10 days from receipt at laboratory <u>Technical and Contract to Analysis</u> : 40 days from extraction	Cool to 4EC ±2EC

^a Individual target compounds are listed in Table 1B.

^b Freezing the sample can extend the holding time; however, 48 hours unfrozen holding time will be considered cumulative.

^c Use Method 5021 for headspace analysis.

^d Use Method 5030 for purge and trap.

^e Use Method 5035 for purge and trap.

^f Diesel analysis does not require acidification; however, acidified samples can be analyzed for diesel.

Data Calculations and Reporting Units:

Calculate the sample results using calibration factors determined according to Sections 7.7.2 of Method 8015B and 7.4.2 and 7.8.1 of SW-846 Method 8000A.

Report water sample results in concentration units of milligrams per liter (mg/L). Report soil sample results on a dry-weight basis in milligrams per kilogram (mg/kg).

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List and Contract Required Detection Limits (CRQL) for SW-846 Method 8015B

COMPOUND	CRQL Water mg/L	CRQL Soil mg/kg
Gasoline - Purge and Trap	0.5	10
Gasoline - Headspace	5.0	5
Diesel	0.5	10

Table 2. Summary of Calibration Procedures for Total Petroleum Hydrocarbons (TPH) as Gasoline and Diesel by SW-846 Method 8015B

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for CFs #20%; or, if using a linear calibration curve, a correlation coefficient (r) of ≥ 0.99 for each compound	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each 12 hour time period, after every 10 samples and end of run	%D between calculated amount and nominal amount within $\pm 25\%$	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Retention time evaluation CCV standards	Each analysis of CCV standards	$\pm 3 \times$ the SD of the avg ICAL RT for surrogate, See footnote ^c for GRO and DRO	1. Re-calibrate and verify 2. Re-analyze samples back to last good IPC

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b Gasoline Range Organics (GRO): C_6 - C_{10} ; Diesel Range Organics (DRO): C_{10} - C_{28}

^c Establish and report retention time (RT) windows for GRO and DRO as described in Section 7.4 of Method 8015B and 7.6 of Method 8000B. Calculate RT windows for GRO and DRO based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component. Determine RT windows for surrogate compounds as $\pm 3 \times$ the standard deviation (SD) of the average ICAL RT for each analyte.

Table 3. Summary of Internal Quality Control Procedures for Total Petroleum Hydrocarbons (TPH) as Gasoline and Diesel by SW-846 Method 8015B

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	<u>Gasoline</u> : One per each 12 hour time period and after unusually concentrated samples <u>Diesel</u> : One per Batch or SDG ^a (1 per 20 samples minimum) and for each instrument used	< CRQL for each compound	1. Investigate source of contamination and document corrective action measures 2. Re-extract and re-analyze all samples processed with a method blank that is out-of-control
Surrogate Spike ^b	Every sample and MB at midpoint of calibration range	<u>Water Samples</u> : 75-125% of expected value <u>Soil Samples</u> : 60-125% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	65-135% of expected value; #30 RPD between MS and MSD	1. Report in case narrative
Laboratory Control Sample (LCS)	<u>Gasoline</u> : one each 12-hour period; <u>Diesel</u> : one per Batch or SDG	80-120% of expected value	1. Re-extract and re-analyze all samples processed with out-of-control LCS

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Surrogates for GRO: 4-bromofluorobenzene and 1,1,1-trifluorotoluene;
Surrogate for DRO: hexacosane or ortho-terphenyl

Dilute and reanalyze samples with one or more analytes at concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.